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Quality effects caused by torrefaction of pellets made from Scots pine

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ABSTRACT

The purpose of the study was to investigate the influence of torrefaction on the quality of Scots pine pellets. Pellet samples were torrefied at 230, 250 and 270 °C for 1 h in nitrogen atmosphere. Higher heating value (HHV) was increased from 18.37 MJ kg^{-1} to 24.34 MJ kg^{-1}. The energy to crush a pellet by mechanical compression was determined using a material tester and results showed a rapid decrease before torrefaction temperature reached 250 °C. Slightly further decrease was observed when increasing the temperature up to 270 °C. The strength loss was confirmed by determining the energy required for grinding the pellet samples in a bench scale disc mill. Particle size distribution measurements after grinding indicated a significant increase of small particles (diameter < ca. 1 mm) for torrefied pellets at a torrefaction temperature of 230 °C and further increase of temperature resulted in steep decrease of large particles (diameter > ca. 2 mm). To further analyze the effect on strength, the mechanical durability of pellets was tested according to wood pellet standards, EN 15210-1. The results have shown a good correlation between pellet durability and compression strength, and indicated that the pellet durability can be estimated based on compression strength data of about 25 pellets.

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1. Introduction

Wood pellets are the only solid biofuels which have a global market. The global production of fuel pellets from biomass was estimated to be about 13 million tons in 2009 with strong growth rates, and pellet consumption in Europe is expected to reach 50 million tons per year by 2020 [1]. Since pellets are very suitable for long-distance transport, the pellet price is sensitive to the transportation cost. In case of Canada, data from 2004 has shown that local transportation, storage and oversea shipment (for example to Rotterdam harbor) cost 3.2 € GJ^{-1}, which is more than half of the market price of pellets (5.9 € GJ^{-1}) [2]. As a consequence, high energy density combined with easy handling properties, i.e. low dust formation, is desired when producing pellets. The energy density of wood pellets can be increased by means of torrefaction, which is a mild temperature (200-300 °C) pretreatment process of biomass in an inert atmosphere [3]. During the process, the biomass looses water and a proportion of its volatile content, becoming dry, brittle and darker in color. Torrefied biomass is more hydrophobic, has a higher calorific value and is easier to grind when compared to untreated biomass [4,5]. Torrefaction can reduce the energy consumption required for size reduction up to 70–90% compared to untreated biomass [5]. Nevertheless, it has been shown earlier that torrefaction can lower the pellets mechanical properties, which might result in problems during transportation and handling. Therefore it is important to investigate the changes of mechanical properties induced by torrefaction at different temperatures. European standards such as the ENPlus set high demands for the pellets mechanical properties i.e. durability and dust formation. According to a defined test protocol the pellet durability may not fall below 97.5% [6], meaning that less than 2.5% dust may be formed during the test. Several studies [5,7–10] have been made investigating the pelletizing properties of torrefied biomass. Two studies made by Stelte et al. [10] and Gilbert et al. [9] indicate that pellet production from torrefied biomass can be challenging and can result in problems during processing and pellet quality. It was shown that torrefaction of biomass increases the friction in the press channel of a pellet mill and that the manufactured pellets are more brittle and less strong compared to conventional pellets. However, no studies have been reported so far for pellets that are torrefied subsequent to pelletization.

Wood pellets made from untreated biomass have high mechanical properties which arise from the thermal softening of lignin, its subsequent flow and the formation of what can be referred to as an "entanglement network of molten polymers" [11]. The interpenetration of lignin polymer chains results in strong bonds upon cooling and solidification of the pellet. The idea behind the present study was to investigate whether these strong bonds outlast the torrefaction process and whether this might be a feasible method to produce pellets of a

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high mechanical stability and high energy density. In the present work, Scots pine pellets were torrefied at 230, 250, and 270 °C with residence time of 1 hour under nitrogen atmosphere. Higher heating value (HHV) was determined using a bomb calorimeter. The mechanical properties were determined by compression testing, milling in a bench scale disc mill and durability testing according to a standard protocol.

2. Material and methods

2.1. Torrefaction of pellets

Scots pine (*Pinus sylvestris*, L.) pellets, 6 mm in diameter were supplied by a commercial pellet factory. Samples were dried in the oven at 104 °C for 24 h, and subsequently placed in an air tight metal reactor (15×31×10 cm) with a gas in and outlet. The reactor was placed in an oven (type S 90, Lyngbyovnen, Denmark) and heated up to the desired torrefaction temperature. The heating rate programmed for the oven was 6 °C min^{-1}. Nitrogen flow through the reactor was adjusted to 0.5 L min^{-1} to create an inert atmosphere. A thermocouple placed in the middle of the reactor was used for temperature control of the oven. The residence time of the torrefaction process was started when the thermocouple inside the reactor has reached the set temperature. After one hour at the desired temperature the oven was shut down and the reactor was allowed to cool down. Torrefaction was carried out at 230, 250, and 270 °C with residence time of 1 hour. The total weight loss (TWL, %) was determined based on Eq. (1), where m_b is the mass (kg) of original sample before drying and m_a is the mass (kg) of residues after torrefaction.

$$TWL = 100 \times \left(1 - \frac{m_a}{m_b}\right) \tag{1}$$

2.2. Heating value

A bomb calorimeter (6300, Parr Instrument Company, USA) was used to determine the higher heating value (HHV, MJ kg^{-1}). Initially, the calorimeter was calibrated using benzoic acid tablets. Samples were prepared by grinding in a commercial coffee grinder (Kenia, Mahlkönig, Germany); 1 g of material was placed in the crucible and fired inside the bomb calorimeter using an ignition wire in the presence of oxygen. The measurements were repeated 2 times. The determination of energy loss was based on Eq. (2).

$$\begin{aligned} Energyloss &= \frac{m_b \times HHV_{ref} - m_a \times HHV}{m_b \times HHV_{ref}} \times 100 \\ &= \left[1 - \frac{HHV}{HHV_{ref}} \times \left(1 - \frac{TWL}{100}\right)\right] \times 100 \end{aligned} \tag{2}$$

Where HHV_{ref} is the heating value of untreated pellets in the unit of MJ kg^{-1}.

Table 1
Mass fraction of hemicelluloses, cellulose and lignin in oven dried pellets and pellets torrefied at 300 °C (dry and ash free basis).

	Lignin	Cellulose	Hemicellulose	Ash
Oven dried wood pellet	32.5	39.5	20.1	0.7
Wood pellet torrefied at 300°C	100.7	0.1	0.0	0.9

2.3. Biomass composition

The content of lignin, cellulose and hemicelluloses was determined according to the ASTM standard E 1758-01 [12], and Kaar et al. [13]. A representative sample smaller than 1 mm was first made soluble in strong acid (72% H_2SO_4) at room temperature; and then it was hydrolyzed in dilute acid (4% H_2SO_4) at 121 °C by autoclavation. Hemicelluloses and cellulose contents were determined by HPLC analysis of liberated sugar monomers. Klason lignin content was determined based on the filter cake (the residues from the strong acid hydrolysis), minus the ash content of the filter cake determined by 550 °C incinerating for 3 h.

2.4. Pellet compression energy

Pellets were tested individually by compressing between two smooth metal platens in a material tester (AG250kNx, Shimadzu, Japan). The pellet was laid horizontally on the lower platen, and the upper platen was then moved down to compress the pellet. The upper platen was mounted on a load cell, and run with a compression speed of 25 mm min^{-1}. The upper platen traveled to 4.5 mm above the lower platen before returning to its initial position. As the pellet diameter was 6 mm it means that the pellets were compressed 1.5 mm, which caused an irreversible deformation (crushing). To quantify the pellet strength, the force and the corresponding position (displacement) of the upper plate was logged (10 ms logging interval). The data was used to calculate the energy required for compressing the pellet. 25 pellets were used for each sample. Prior to compressing, the length and pellet mass were determined.

2.5. Grinding energy

Energy consumption for grinding pellets was determined using a commercial coffee grinder (Kenia, Mahlkönig, Germany) with a screw conveyer feeding system and a disc grinding system, as shown in Fig. 1. The distance between the two separate discs could be adjusted manually and was set to maximum for all tests. The power consumption of the coffee grinder in operation was determined using a wattmeter (THII, Denmark). The meter was connected to a data logging system (NI USB-6009, National Instruments, USA).

Approximately 200 g pellets were manually fed into the feed hopper while the grinder was running. The time required to grind the pellet and the energy used by the grinder were recorded and used for calculating the specific energy required for grinding. The idling energy was measured before the material was introduced. The specific energy required for grinding was determined by integrating the area under the power

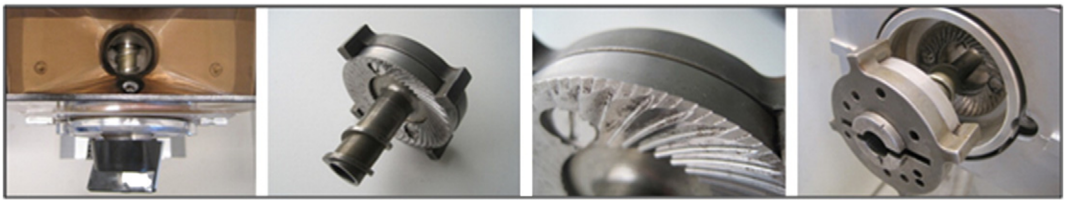

Fig. 1. Picture of the inside of the coffee grinder.

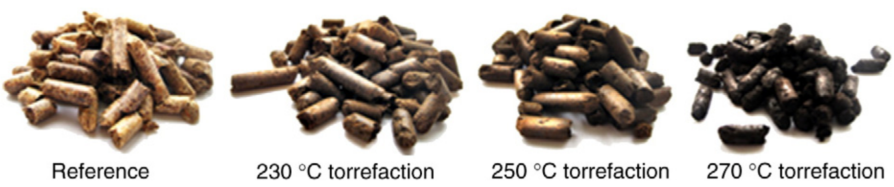

Fig. 2. Images of untreated Scots pine pellets and pellets torrefied at different temperatures for 1 h.

demand curve for the total time required to grind the sample minus the idling power [14,15]. The energy reduction during grinding was calculated according to Eq. (3):

$$energyreduction = \frac{\Delta E}{E_{ref}} \times 100 = \frac{E_{ref} - E_{samp}}{E_{ref}} \times 100 \quad (3)$$

where E_{ref} is the specific energy (J g^{-1}) required for grinding untreated pellets, E_{samp} is the specific energy (J g^{-1}) required for grinding pellets torrefied at different temperatures.

Particle size distribution was calculated based on sieve separation of the obtained biomass fraction using a sieve shaker (Retsch, Germany) with nine different sieves (mesh size of 75, 125, 250, 500, 1000, 1400, 2000, 3150, 5000 μm). The sieve shaker was run for 40 minutes and the weight of the individual fractions was determined subsequently.

2.6. Standard durability test

The pellet durability was determined according to the EN 15210-1 standard [16], also known as tumbling can test, where a defined mass of pellets is filled into a metal container and exposed to impacts by rotating the containers for a defined period of time. The amount of fines formed during this test, is used to quantify the pellet durability. Prior to the testing, pellets were sieved through a 3.15 mm screen (round holes) to remove fines and dust from the samples. The amount of dust is referred to as 'dust in sample', and quantified as the difference in weight of the pellet sample before and after sieving, in percent of the sample before sieving. 500 g dust free pellets were then loaded into the chamber of the standard durability tester and exposed to 500 rotations within a time interval of 10 min. The amount of fines formed during the test was determined by sieving the treated sample again through the 3.15 mm screen and determination of the weight difference before and after sieving. The durability value was calculated as the mass fraction of dust free pellets after the treatment in the pellets that was loaded into the tester.

3. Results and discussion

3.1. Torrefaction

The composition of the Scots pine pellets used in this study is listed in Table 1. Images of untreated pellets (noted as 'reference') and pellets torrefied at different temperatures are shown in Fig. 2. The pellets became darker with an increasing torrefaction temperature and the pellet surface has lost its shine and smoothness, especially for the pellets torrefied at 270 °C. The color change of thermally modified wood was studied by González-Peña and Hale [17], and they concluded that color change is linked to changes in the acid-insoluble lignin substance rather than in the carbohydrate fraction.

Results for the HHV, ratio between HHV of reference pellets and torrefied pellets, TWL, corresponding energy loss are listed in Table 2. The results indicate that in case of weight based charging of transportation fees for pellets about 10–25% of the cost can be saved by transporting the same amount of heating value of torrefied pellets compared to regular wood pellets. To account for the correlation between energy loss and weight loss, a second-order polynomial regression has been made and is shown in Fig. 3. A good correlation was found between energy loss and weight loss. By increasing torrefaction temperature from 250 to 270 °C, both weight loss and energy loss increased sharply. Based on our previous studies of torrefaction effects on spruce [10] and wheat straw [18] , this is mainly due to the extensive thermal degradation of hemicelluloses and partial degradation of cellulose at elevated temperatures. Thus, in order to preserve energy in the pellets, a torrefaction temperature of about 250 °C might be a critical upper limit.

3.2. Compression energy

Pellet before and after compression in the universal materials test are illustrated in Fig. 4.

Fig. 5 shows force/displacement curves for compression of 25 reference pellets. The plots illustrate the position (Displacement, mm) of the compression platen from 2 mm above the pellet to 5.5 mm, from where the platen returned to its starting point. It can be seen that the platen meets the pellet at position 4 mm, where the force increased as the pellet was crushed. The energy (in J) required for compressing the pellets to 75% of the initial diameter (4.5 mm vs. 6 mm) was calculated based on the area under the plots. Since the pellets had different lengths, the compression energy of the distinct pellets was different. To account for this, linear regressions of strength (in J) vs. length were made (see Fig. 6). It can be seen that the longer the pellet, the higher the energy required for compressing. The regression parameters were used to predict the strength of a model pellet with the length of

Table 2
Data of higher heating value (HHV), total weight loss (TWL on wet basis), energy loss, and ratio between HHV of untreated pellets and pellets torrefied at different temperatures for 1 hour.

Temperature	HHV, MJ kg^{-1}	TWL[a] (w.b.), %	Energy loss, %	HHV$_{ref}$/HHV
Reference	18.37 (0.01)[b]	0	0 (0.05)	1
230 °C	20.42 (0.02)	14.8	5.30 (0.10)	0.90
250 °C	21.35 (0.08)	21.3	8.54 (0.35)	0.86
270 °C	24.34 (0.03)	41.9	23.02 (0.09)	0.75

[a] Pellet samples had a moisture content of 9.8% (w.b.) determined by drying at 104 °C for 24 hours.
[b] Number enclosed in parenthesis are standard errors of the mean values listed.

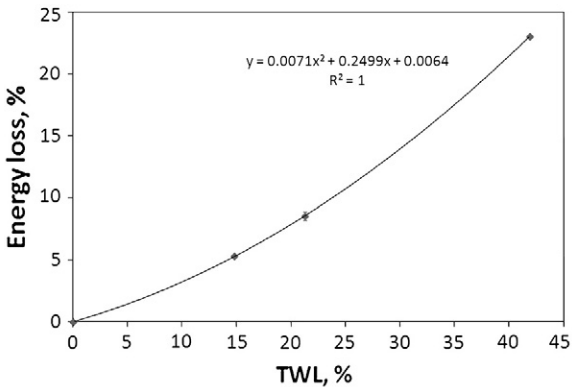

Fig. 3. Energy loss vs. total weight loss (TWL) during drying and torrefaction of pellets torrefied at different temperatures.

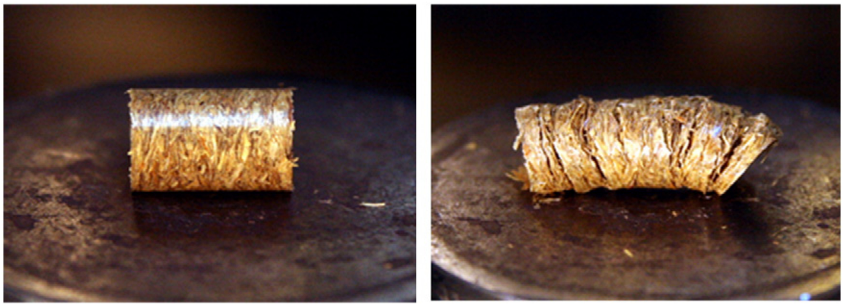

Fig. 4. Pellet before and after compression in the universal material machine.

15 mm. This strength of compressing single pellet was further converted to weight based strength by the mass. Results are presented in Table 3.

3.3. Grinding energy

Fig. 7 shows the relationship between the specific energy requirement of grinding pellets and the total weight loss (TWL). Results of the single pellet compression strength are also presented in this figure. Both results show the similar trend of energy decreasing when the TWL increases. Fig. 8 plots energy reduction against pellets' energy loss, which is listed in Table 2. It can be seen that about 73% of the energy used in the grinding process can be saved by losing 9% of the pellets' heating value due to torrefaction, but further increase of the pellets’ energy loss does not reduce the grinding energy a lot. However, the absolute value of the thermal energy lost from the pellets ($1653\ \mathrm{J\,g^{-1}}$) is much higher than the electrical energy saved during grinding ($19.6\ \mathrm{J\,g^{-1}}$) in this case. The fraction of grinding energy saved due to torrefaction in torrefied pellets’ HHV was also plotted in Fig. 8 for different torrefaction temperatures. Since the energy consumption during grinding depends on the end particle size, a higher absolute value of energy saving can be expected with decreasing particle size, when fine grinding is chosen.

Mass fractions of the particles retained on each test sieve in relation to the geometric mean diameter of the particles on each sieve are shown in Fig. 9. An obvious increase (8% compared to reference sample) of small particles (about 1 mm) occurs already at a torrefaction temperature of 230 °C. Further increase of torrefaction temperature (from 230 to 270 °C) resulted only in slight increase of small particles (+ 5%), but steep decrease of big particles (- 10%).

3.4. Standard durability test

The results from the standardized mechanical durability test are presented in Fig. 10. It can be seen that the torrefaction resulted in pellets with more dusts and lower durability when exposed to mechanical loads. And this effect increases with increasing torrefaction temperature.

Fig. 11 illustrates the correlation between the durability and the compression energy. It is seen that an exponential function fits the correlation well. This indicates that if the compression energy is known, a reasonable estimate for the durability can be made and vice versa. It must also be noted that the analysis was based on pellets with a diameter of 6 mm only, which means the diameter parameter has not been taken into account, and that the present parameters may not fit i.e. 8 mm pellet. However in this case, if pellets of 15 mm lengths have compression strength higher than 0.6 J ($1.53\ \mathrm{J\,g^{-1}}$ pellet

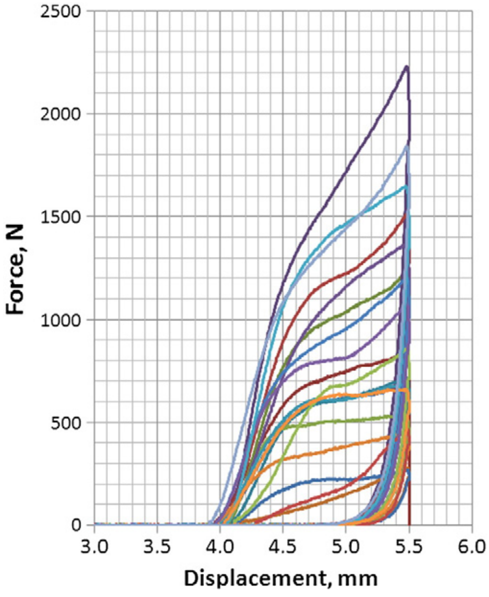

Fig. 5. Plot illustrating the compression strength analysis of 25 reference pellets.

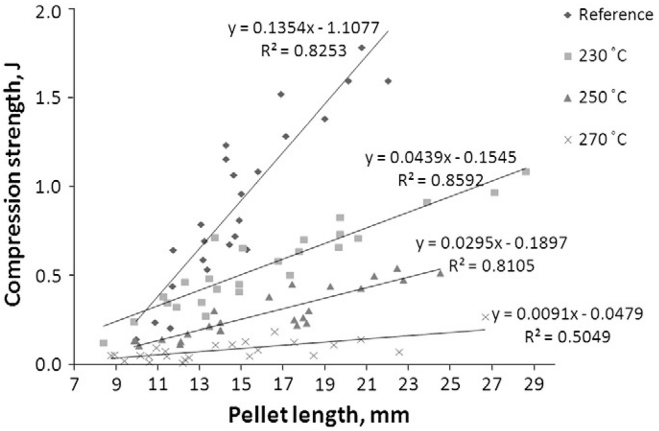

Fig. 6. Linear regressions of compression strength as y-axis (in J) vs. pellet length as x-axis (in mm).

Table 3
The compression strength of a model pellet (15 mm long) based on the linear regressions in Fig. 6.

Sample	Strength, $\mathrm{J\ pellet^{-1}}$	Strength, $\mathrm{J\ g\text{-}pellet^{-1}}$
Reference	0.92 (0.20)[a]	2.25 (0.50)
230 °C	0.50 (0.09)	1.34 (0.25)
250 °C	0.25 (0.06)	0.76 (0.19)
270 °C	0.09 (0.04)	0.35 (0.18)

[a] Number enclosed in parenthesis are standard errors of predicted strength from the regression parameters in Fig. 6.

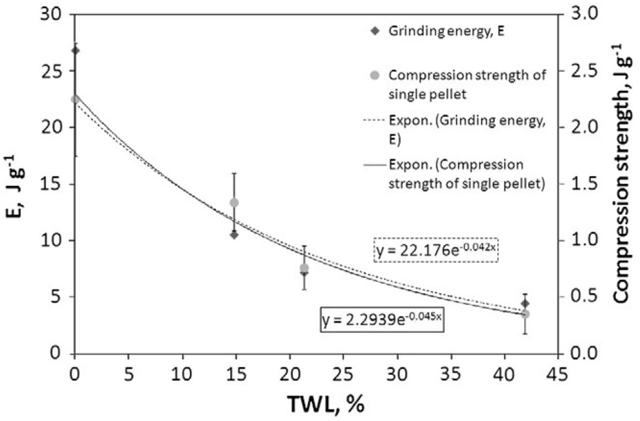

Fig. 7. The specific energy required for grinding pellets (E, J g^{-1}) and the compression strength of single 15-mm-long pellet (J g^{-1}) vs. total weight loss (TWL) of pellet samples after torrefaction.

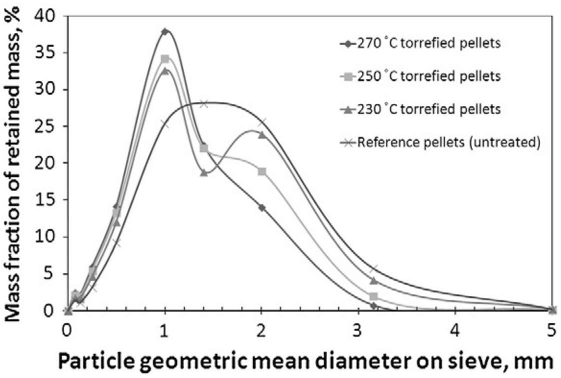

Fig. 9. Particle size distribution of fines after the energy consumption measurement.

according to Table 3), it is likely the batch from which they are taken has durability higher than 97.5%.

4. Conclusion and discussion

The study has shown that higher torrefaction temperature resulted in higher weight and energy loss. A steep increase of total weight loss (from 21% to 42%) and energy loss (from 9% to 23%) occurred when the torrefaction temperature was increased from 250 to 270 °C. In order to preserve energy in the pellet, torrefaction temperatures higher than 250 °C should be avoided. However, if shorter residence time is used in production, torrefaction temperature higher than 250 °C could be relevant.

Both single pellet compression energy and energy required for disc mill grinding showed a similar exponential decrease when TWL increases. Rapid decreases happened in the first 20% TWL; however reduction was less when TWL increased from 20% to 40%. The particle size distribution after disc mill grinding showed that an obvious increase of small particles with diameter of about 1 mm happened already at torrefaction temperature of 230 °C, whereas further increase of temperature resulted in only slight increase of small particles but extensive decrease of large particles with diameter of about 2 mm.

The pellet durability showed negative relationship with torrefaction temperature, and good correlation was shown among pellet durability, the compression strength measured in a universal materials tester, and the energy requirement for grinding. It means that compression strength of single pellet can be used as a product quality control method to predict the durability of the whole batch pellets and the energy use in grinding and vice versa.

In order to produce pellets (with diameter of 6 mm) with durability higher than 97.5% as required by ENPlus, compression strength of 0.6 joule per pellet with length of 15 mm (or 1.53 joule per gram pellet with length of 15 mm) is needed, and grinding energy of about 15.3 J g^{-1} is expected. At this condition, energy reduction in grinding compared with untreated pellets is 43% (11.6 J g^{-1}), and the energy loss due to the torrefaction of pellets is about 4% (730 J g^{-1} based on original mass). However, the higher heating value (HHV) is increased by about 1600 J g^{-1} from 18.4 MJ kg^{-1} for reference pellets to 20.0 MJ kg^{-1}, which corresponds to a torrefaction temperature of a bit lower than 230 °C with residence time of 1 h.

On the other hand a previous work [10] has shown that pellets pressed from torrefied spruce increase significantly in length after pelletization, which indicates worse quality of inter-particle bonding with correlation to higher torrefaction temperatures. The pellets produced in this study (put the torrefaction step after the pelletization step) exhibit better durability, no spring back effect or disintegration was observed for pellets even torrefied at 270 °C. Similar conclusions of low durability and low mass density were also drawn for pellets made from pine wood torrefied at 300 °C by Reza et al. [19] in a recent study, although they succeeded making mechanically durable pellets from hydrothermally carbonized pine wood without additives. However, there is lack of results for pellets made from biomass torrefied at temperatures below 250 °C. Furthermore, all pellets made by pelletizing torrefied biomass reviewed here [9,10,19] are from single pellet press, which works in a different way compared to commercial

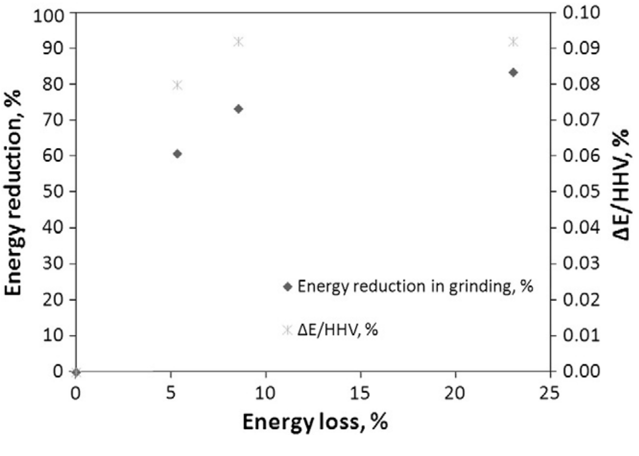

Fig. 8. Energy reduction during grinding and the fraction of reduced grinding energy (ΔE) in sample's higher heating value (HHV) vs. pellets' energy loss due to torrefaction.

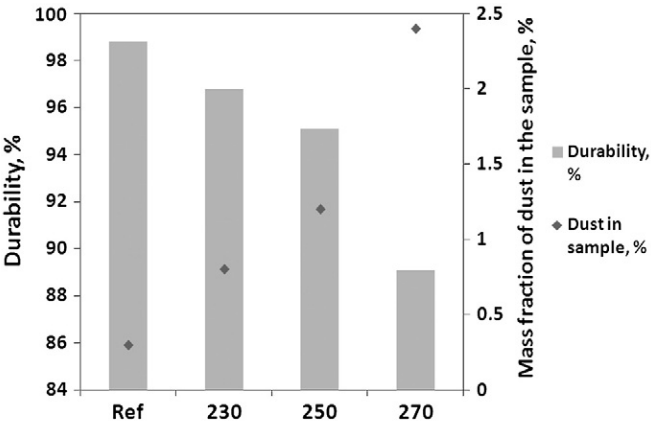

Fig. 10. The results from the standard durability test and mass fraction of dust in the sample prior to the test.

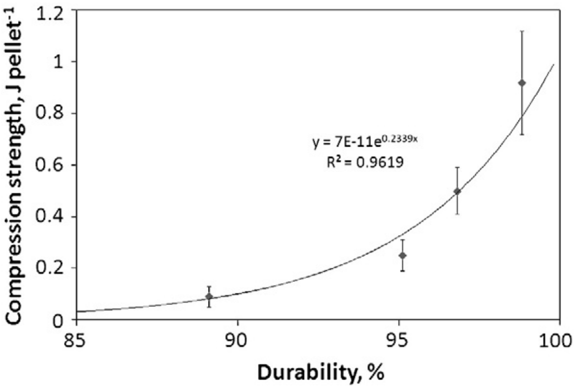

Fig. 11. Plot of compression strength of single 15 mm-long pellet (J pellet^{-1}) vs. durability. The fit of an exponential function is illustrated.

pellet mills. In fact, a variety of pellets made from torrefied biomass are available in markets and they possess very good durability, which means the pellet quality can still be enhanced by adding additives or improving the torrefaction process, for example wet torrefaction.

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